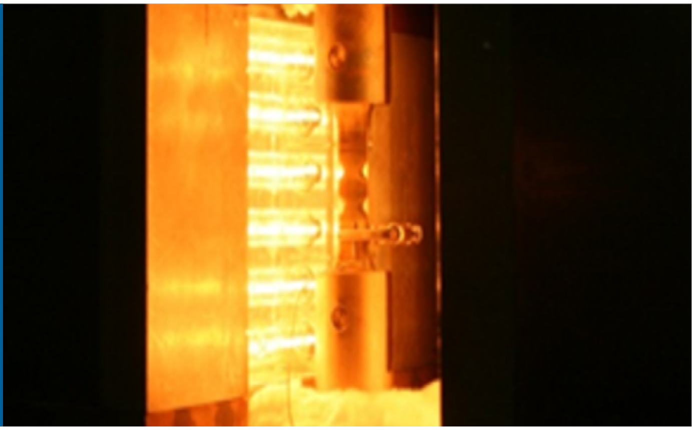
EXPLORATORY RESEARCH: CORE POWERTRAIN MATERIALS

DEVELOPMENT OF HIGH TEMPERATURE SAMPLE ENVIRONMENT FOR ADVANCED ALLOY CHARACTERIZATION USING HIGH-ENERGY X-RAY TECHNIQUES

Project ID: mat179



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X-ray Science Division, Argonne National Laboratory

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Annual Merit Review
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OVERVIEW

Timeline

- Project start: March 2019
- Project end: March 2020
- 90% complete

Budget

- FY19 = $100 K (DOE)

Project Partners

- Applied Materials Division, Argonne National Laboratory

Barriers

- Performance: Flexible heating device for various material system and specimen geometry. Expand temperature limit up to 1400 ℃ with high heating and cooling rate (>10 ℃ /sec) to probe various part of TTT diagram.

- Thermal stability: Device should have minimal thermal load to the surroundings. Requires high thermal stability to minimize motion blur and improve image quality

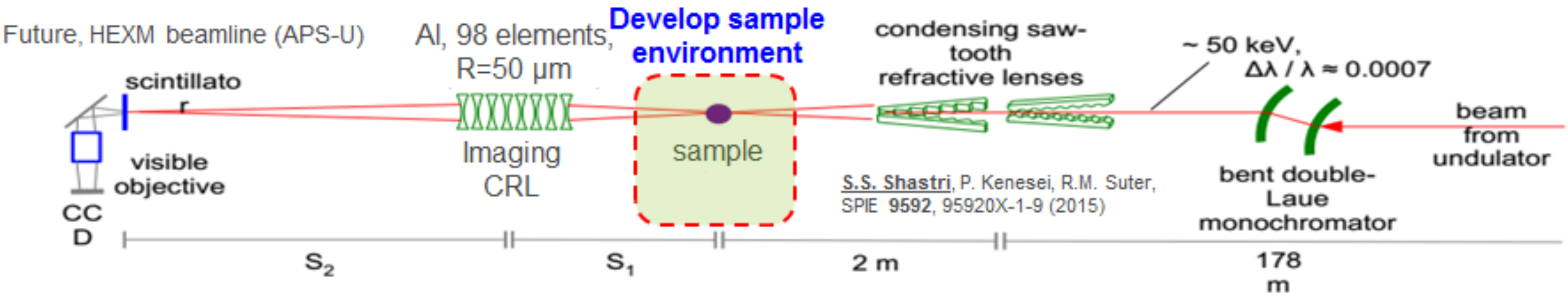
RELEVANCE

Develop characterization capability to support alloy development research

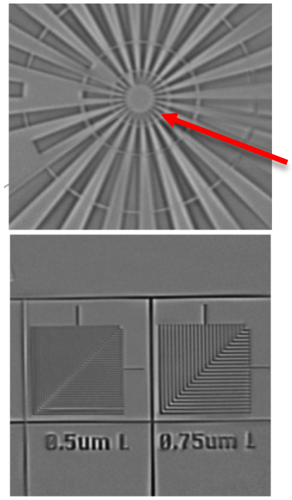
- Motivation:
  - A strong and growing interest to access the most advanced synchrotron X-ray techniques for in-situ material characterization.
  - To understand material behavior under in-service conditions requires information at elevated temperature
- Objectives:
  - Develop flexible high temperature sample environment that allows μm stability for **high resolution**, **zoom-in/out** imaging and diffraction techniques
  - Provide improved sample environment accessibility and flexible sample environment, avoid duplicate effort among community, enhance scientific productivity, and enable expansion of in situ testing capabilities.
- Impact
  - Facilitate beamline access for advance powertrain materials research
  - Provide the powertrain community with access to a wide range of X-ray techniques at the APS.

APPROACH

Full-field X-ray Imaging at Elevated Temperature



- Locate region of interest in the bulk sample with regular μ-tomography (resolution 1~2 μm)
- Use x-ray lens view internal structure of polycrystalline materials such as voids, cracks and inclusions. (resolution ~0.5 μm [now], ~0.1 μm [future])
- Proposed project adds high temperature environment to the high energy x-ray beamline to study material under in-service condition



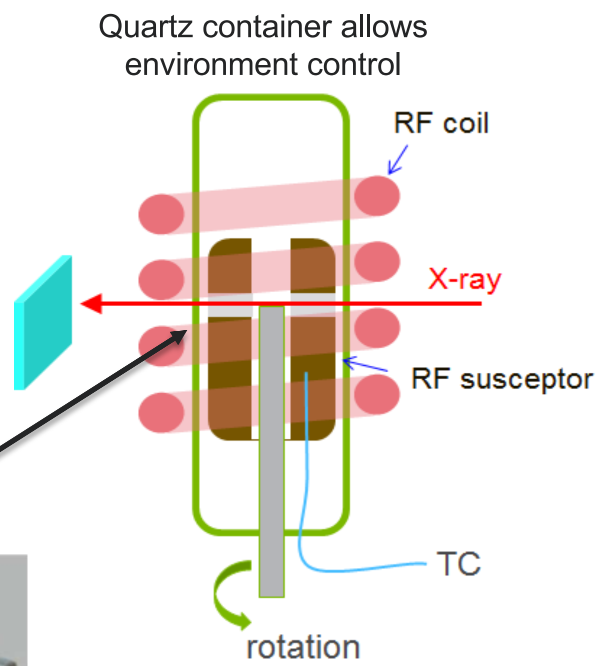
0.5 μm finest feature

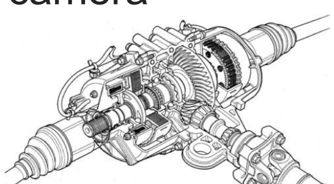
Argonne NATIONAL LABORATORY

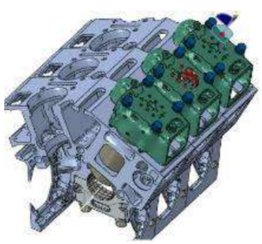
APPROACH

Rapid heating/cooling with high thermal stability

- Induction based heating
- Flexible heating geometry with custom coil
- High heating and cooling rate (> 20°C/sec)
- Heat only the specimen, minimize heat load to the environment. Enables µm level imaging.
- Use susceptor to increase temperature uniformity
- Closed-loop temperature control.
- Tempera monitor by thermocouple and thermo camera



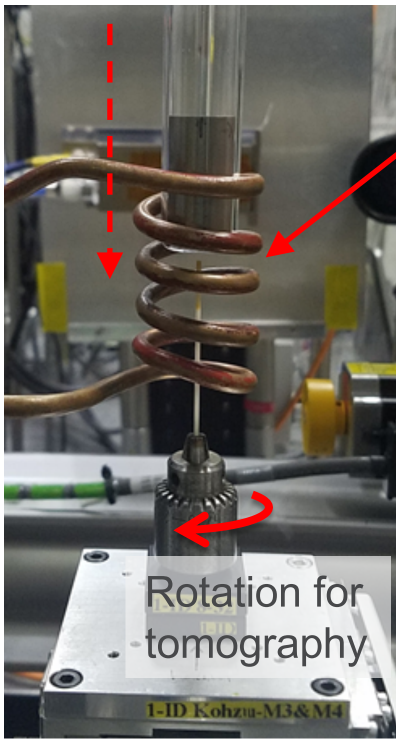

Drive shaft material


Engine material

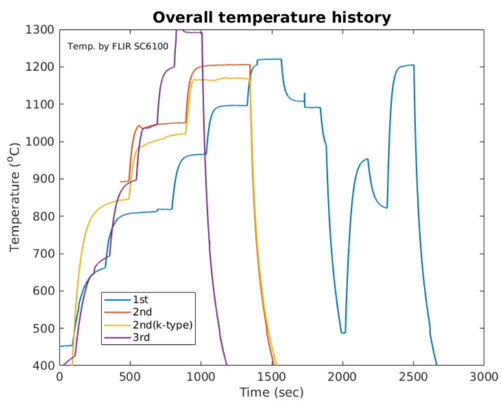

Crankshaft material

Technical Accomplishments

HIGH TEMPERATURE 3D IMAGING SETUP

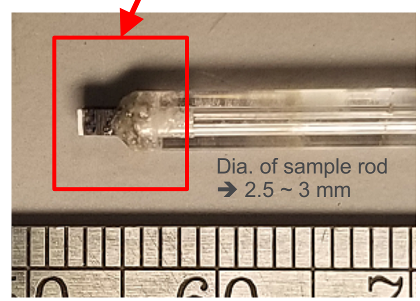


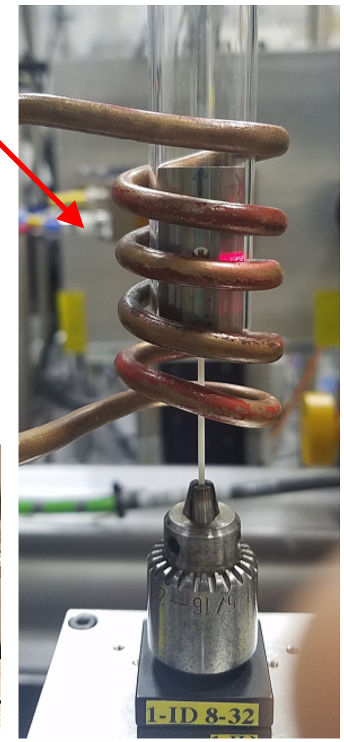
- Susceptor in quartz tube.
- Whole structure moves down to cover sample.
- Sample is placed at center of susceptor.



Device heating/cooling rate >25 °C/sec

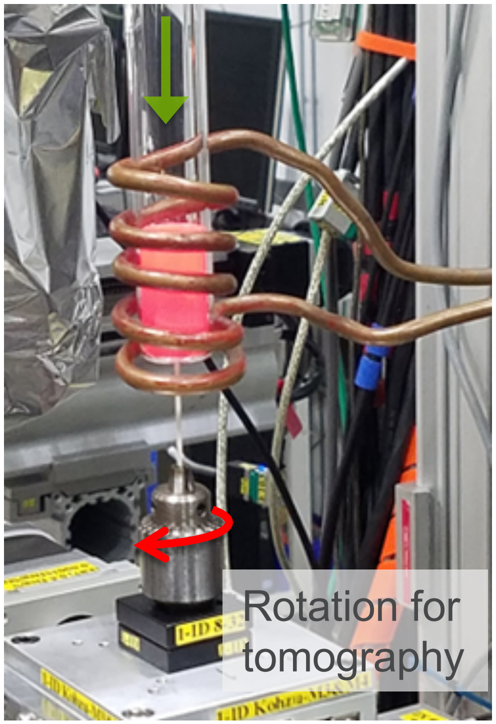
Sample on ceramic(quartz) rod. Glue with high temperature paste



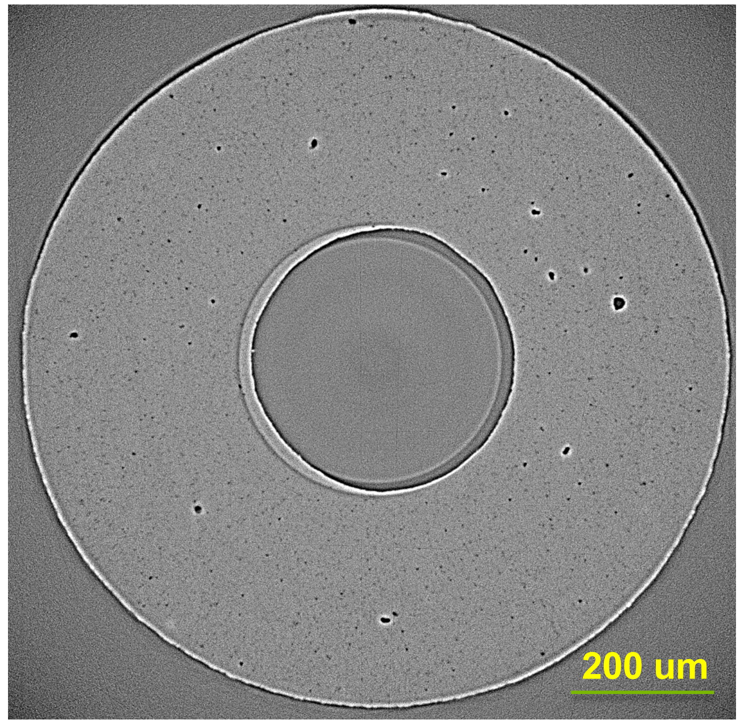


TOMOGRAPHY AT HIGH TEMPERATURE

Flush with Ar during experiment



Tomography Image at 900 °C



PROJECT MILESTONE AND COLLABORATIONS

Milestone

Milestone	Description	Status
System design, development and test	• Design and construct the system • Test in-house without X-ray	• Close loop temperature control system finished and tested • Reach temperature target of 1350 °C with stability ± 5°C
Commission the system to work with existing beamline equipment	• Test the system at the beamline • Conduct x-ray imaging and diffraction experiment and elevated temperature	• Conducted two user experiment • Collect tomography image at 900°C and diffraction at 1150°C

Collaborations

- MINES Saint-Etienne, France (Andras Borbely) – In-situ study of recovery and recrystallization of an AlScZr alloy
- Caltech, CA, USA (Benjamin Herren, Katherine Faber) - Damage Evolution in Ceramic-Matrix Composite/Environmental Barrier Coating Systems

PROPOSED FUTURE RESEARCH*

(*Any proposed future work is subject to change based on funding levels)

- Add and integrate a compact load frame to the system to characterize engineering material under load at elevated temperature.

PROJECT SUMMARY

- Rapid heating and cooling cell for high-resolution, high-energy x-ray imaging and diffraction techniques has been developed.
- The system utilizes the concept of induction heating to enable rapid heating and cooling (> 25°C/sec) capability while minimizing the thermal load to the surrounding to achieve ~μm image resolution.
- Proof of concept experiment has been done in late 2019. In a user’s in-situ high temperature X-ray experiment, samples (<1g) was heated up to 1250 °C for diffraction experiment and 900 °C for tomography experiment.